भारतीय मानक
Indian Standard

IS 5182 (Part 7) : 2021

वायु प्रदूषण मापने की पद्धति

भाग 7 हाइड्रोजन सल्फाइड

(पहला पुनरीक्षण)

Methods for Measurement of Air Pollution

Part 7 Hydrogen Sulphide

(First Revision)

ICS 13.040.20

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August 2021 **Price Group 4**

Air Quality Sectional Committee, CHD 35

FOREWORD

This Indian Standard (Part 7) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.

Hydrogen sulfide gas (H_2S) is a colourless, flammable, toxic, extremely hazardous gas with a rotten egg smell. It is heavier than air. It occurs naturally in crude petroleum and natural gas, and can be produced by the breakdown of organic matter and human or animal wastes. People are primarily exposed to hydrogen sulfide through inhalation. The effects depend on how much hydrogen sulfide has been inhaled and the duration of exposure. Exposure to very high concentrations can quickly lead to death. Repeated exposure to low concentrations has an irritating effect on mucous membranes, eyes and respiratory tract.

Hydrogen sulphide is generally present in the ambient atmosphere at low concentrations. The threshold limit value (TLV) for hydrogen sulfide is 1 ppm for 8 h exposure and 5 ppm for short term exposure (15 min).

This standard was first published in 1973. The first revision has been taken up to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

a) Ferric sulphate has been replaced by ferric chloride;

b) Additional interferences identified and measures for reducing the interferences incorporated; and

c) Details of methodology modified for better elaboration.

In the preparation of this standard, considerable assistance has been derived from the following publication:

Method No. 701, LODGE (J. P.) Methods of Air sampling and Analysis (Third edition), 1989.

There is no ISO standard on this subject.

The composition of the committee responsible for formulation of this standard is given at Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 7 HYDROGEN SULPHIDE

(First Revision)

1 SCOPE

1.1 This standard (Part 7) prescribes a method for the measurement of concentration of hydrogen sulphide (H_2S) in ambient air.

1.2 The range of concentration covered in this method is 6 to 600 µg/m^3 of H_2S in air.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards below.

IS No.	*Title*
265 : 1993	Hydrochloric acid
266 : 1993	Sulphuric acid
376 : 1986	Sodium hydroxide, analytical reagent
1070 : 1992	Reagent grade water
4167 : 1980	Glossary of terms relating to air pollution
2303 (Part 1/Sec 1) : 2012/ISO 719 : 1985	Grading glass: Part 1 Method of test and classification, Section 1 Hydrolytic resistance of glass grains at 98 ºC

3 TERMINOLOGY

The definitions given in IS 4167 shall apply.

4 PRINCIPLE

Hydrogen sulphide is collected by aspirating a measured volume of air through an alkaline suspension of cadmium hydroxide. Sulphide in aqueous alkaline solution has the tendency to rapidly oxidize through contact with air. To prevent this oxidation, cadmium hydroxide is used in the absorbing solution so that cadmium sulphide is precipitated. The collected sulphide is subsequently determined by spectrophotometric measurement of the methylene blue produced by the reaction of the sulphide ions with a strong acid solution of N, N-dimethyl-*p*-phenylenediamine and ferric chloride.

5 RANGE AND SENSITIVITY

5.1 Minimum detectable concentration of hydrogen sulphide is 0.03 µg/ml of the absorbing solution with an overall accuracy of ± 20 percent.

5.2 Beer's law is obeyed up to 2 µg H_2S/ml of absorbing solution. Hence, for ambient concentrations of H_2S above 70 µg/m^3, the sampling period shall be reduced or the volume of absorbing solution shall be increased either before or after aspirating.

6 INTERFERENCES

6.1 The methylene blue reaction is highly specific for sulphide at low concentrations. Reducing agents, such as sulphite retard colour development if present in significant concentrations even in the case of solutions containing several µg S^{2-}/ml. However, upto 40 µg/ml of SO_3^- interference can be overcome by adding 0.1 – 0.3 ml ferric chloride and extending the reaction time to 50 min.

6.2 At levels of ≥ 0.5 µg/ml, nitrite produces a pale yellow colour with the reagents. No interference is encountered when NO_2 of concentrations up to 0.3 ppm is aspirated through a midget impinger containing a slurry of cadmium hydroxide - cadmium sulphide.

6.3 Atmospheric oxides of nitrogen cause interference. They may be avoided by adding sulphamic acid after the sampling is over.

6.4 Ozone at 57 ppb reduces the recovery of sulphide precipitated as CdS by 15 percent. Other possible atmospheric interferences like sulphur dioxide and ammonia are not serious at their normal existing levels of concentrations.

6.5 Cadmium sulphide decomposes significantly when exposed to light. Consequently, the absorber vessel needs to be protected from light.

NOTE — Arabinogalactan may be added to the cadmium hydroxide slurry prior to sampling to minimize photo-decomposition of the precipitated cadmium sulphide.

6.6 When sampling air having H_2S concentration ≥ 7 mg/m^3, the sampling time must be limited to 5 min. Prolonged sampling of high concentration of H_2S may result in deposition of sulphur in the frits of the absorber due to oxidation of sulphide. Such deposition causes a gradual decrease in sampling rate in the absence of a device to maintain constant flow rate.

7 APPARATUS

7.1 Sampling Apparatus

The equipment to be used for sampling consists of a standard impinger of 35 ml capacity, a trap, a flow meter or critical orifice device and a suction pump. The arrangement of the sampling train is illustrated in Fig. 1.

7.1.1 *Impinger* — Glass impinger, 35 ml capacity, 220 mm in length and 26 mm wide. Impinging end of the tube shall be of 1 mm bore size or preferably with fritted disc having porosity of 50 microns or less (*see* Fig. 2A and 2B). These glass impingers shall be made from HGB 1 glass conforming to IS 2303 (Part 1/Sec1).

7.1.2 *Flow Meter* — A rotameter calibrated accurately for the flow range from 0.2 l/min (for long-term sampling) to 2 l/min (for short-term sampling).

7.1.3 *Critical Orifice Device* — This device is used to give a flow of about 1 l/min. Normally hypodermic needles are used as critical orifice. The needles shall be protected by membrane filters. The arrangement is illustrated in Fig. 3.

7.1.4 *Trap* — This may be a membrane filter (*see* Fig. 3) or a glass wool trap (*see* Fig. 1).

7.1.5 *Pump* — It shall be a suction device capable of drawing air into the sampling train at the maximum rate of 10 to 20 l/min provided with a vacuum regulator and a moisture trap. It shall be of light mass, suitable for field work (portable) with continuous rating, self-lubricating and capable of operating at 230 V *ac*, 50 Hz.

7.2 Spectrophotometer — Capable of operating at 670 nm using 1 cm path length cuvette. Cuvette with long path length (2-5 cm) is recommended for improved sensitivity.

8 REAGENTS

All reagents should be of analytical grade.

8.1 Reagent Grade Water — Conforming to IS 1070.

8.2 Sulphuric Acid (H_2SO_4) — Conforming to analytical reagent grade of IS 266.

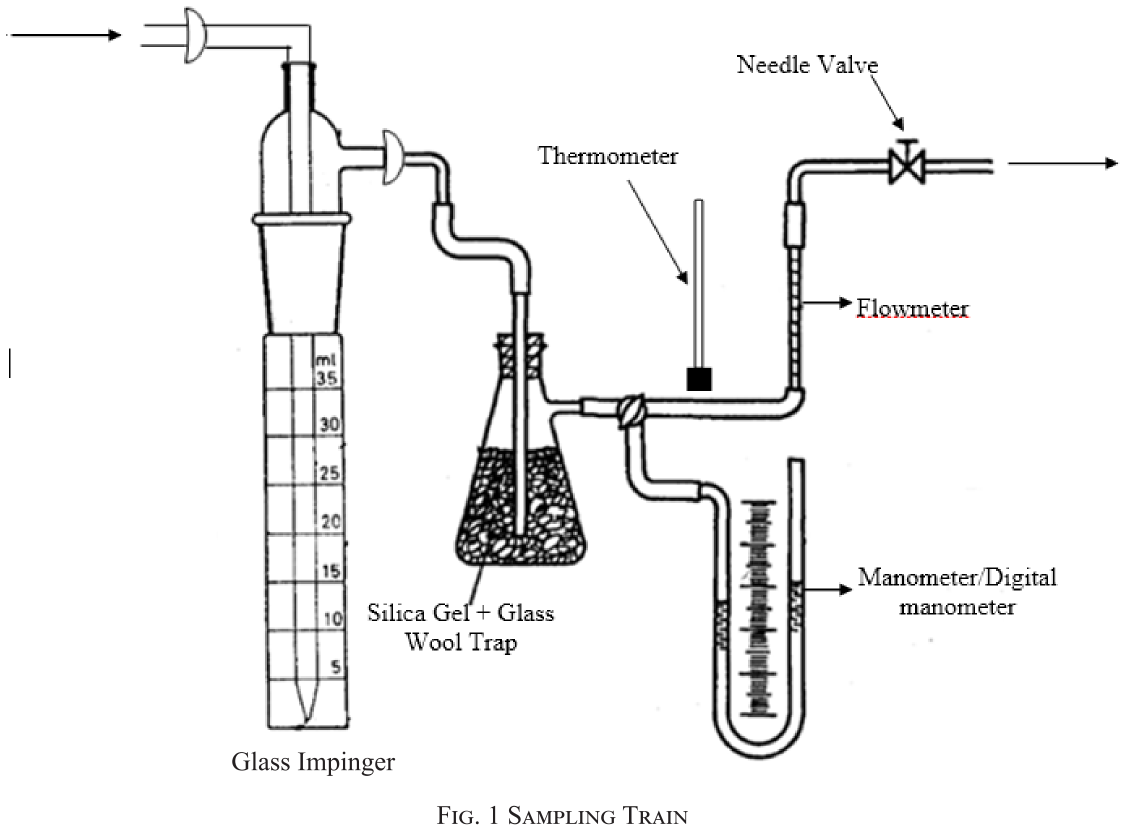
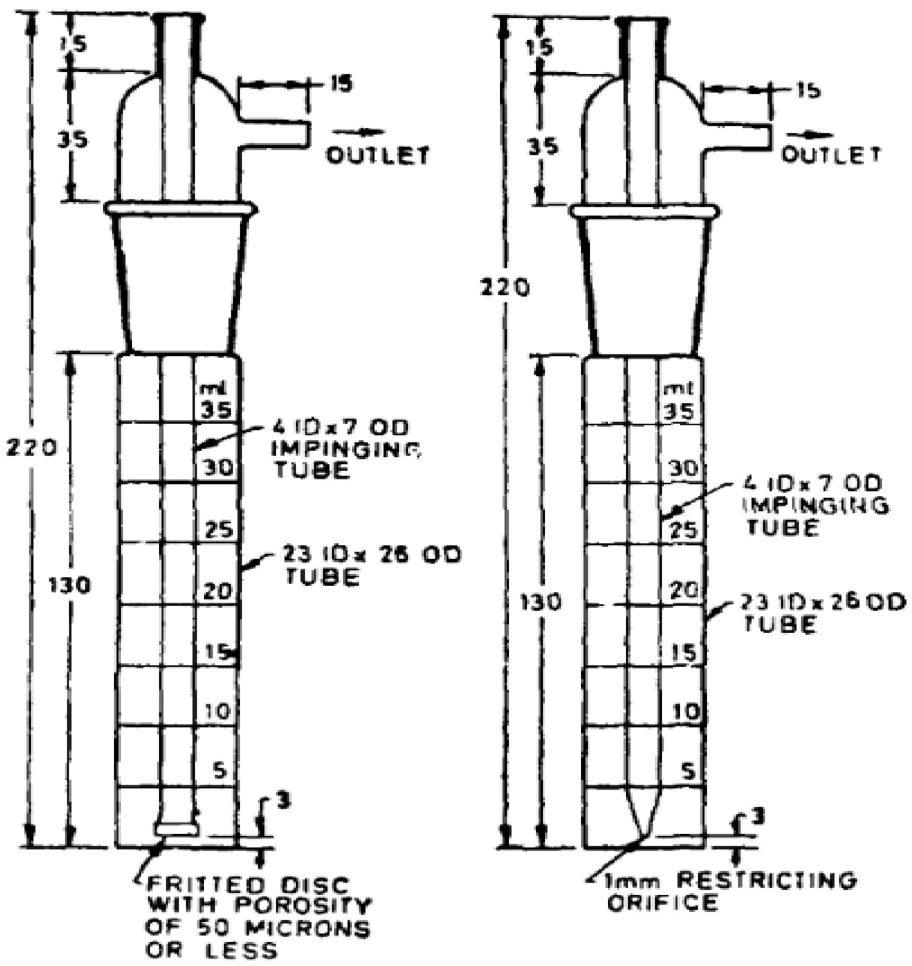


FIG. 1 SAMPLING TRAIN



2A FRITTED IMPINGER   2B STANDARD IMPINGER

FIG. 2 GLASS IMPINGERS

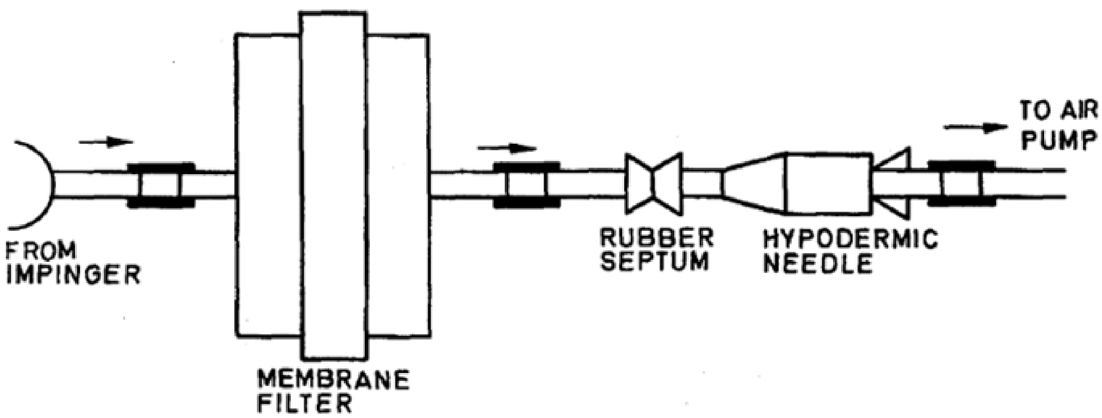


FIG. 3 CRITICAL ORIFICE FLOW DEVICE

8.3 Hydrochloric Acid (‘HCl) — Conforming to analytical reagent grade of IS 265.

8.4 Sodium Hydroxide (NaOH) — Conforming to IS 376.

Amine-sulphuric Acid Stock Solution — Add 50 ml concentrated sulphuric acid slowly to 30 ml distilled water with continuous stirring and allow to cool. Dissolve 12 g N, N-dimethyl-p-phenylenediamine dihydrochloride in the sulphuric acid solution and stir till it is completely dissolved. Store the stock solution in fridge.

8.5 Sulphuric Acid, 50 Percent (*v/v*)

Add 500 ml concentrated sulphuric acid slowly to 400 ml distilled water with continuous stirring and allow to cool. Quantitatively transfer to a 1 litre volumetric flask and make up to the mark.

8.6 Amine-sulphuric acid Standard Solution

Dilute 25 ml of the stock solution in **8.4** to 1 litre with 50 percent sulphuric acid (**8.5**).

8.7 Ferric Chloride Solution, 3.7 M

Dissolve 100 g ferric chloride hexahydrate ($FeCl_3.6H_2O$) in 30 ml water and add 9 ml of concentrated hydrochloric acid. Make up the volume of the solution to 100 ml.

8.8 Ammonium Phosphate, 40 Percent (*w/v*)

Dissolve 400 g of diammonium phosphate in water and make upto 1 litre in a volumetric flask.

8.9 Absorbing Solution

Dissolve 4.3 g of cadmium sulphateoctahydrate ($CdSO_4.8H_2O$) and 1.8 g sodium hydroxide in two separate 250 ml volume water. Add the two solutions to each other and dilute to 1 litre. Shake the resultant suspension vigorously before removing an aliquot. Prepare fresh every 3rd day. The *p*H of the absorbing solution should be maintained at 10 for better results.

8.10 Stock Sodium Sulphide Solution

Dissolve 1.2 g of sodium sulphide ($Na_2S.9H_2O$) in 1 litre of pre-refrigerated distilled water and stopper it. Since the solution is very unstable, it should be prepared just before use. It should be stored in a refrigerator. The approximate concentration of the sulphide solution will be 160 µg S^{2-}/ml. The exact concentration must be determined by iodometric titration using sodium thio-sulphate solution prior to further dilution (*see* **9.4.1**).

8.11 Dilute Standard Sulphide Solution

Take 10 ml of the stock sodium sulphide solution (**8.11**) and dilute it 100 times to bring the volume to 1 litre using freshly boiled and cooled distilled water. Keep the freshly boiled water under an oxygen-free nitrogen atmosphere during cooling. This sulphide solution is unstable. Therefore, prepare this solution immediately prior to use. This diluted solution will contain approximately 1.6 µg S^{2-}/ml.

8.12 Standard Iodine Solution (0.025 N)

Dissolve 10 to 12.5 g potassium iodide (KI) in a little distilled water and add 1.6 g re-sublimed iodine. After iodine has dissolved, dilute to 500 ml with distilled water.

8.13 Standard Sodium-thiosulphate Solution (0.025 N)

Dissolve 6.205 g sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$) in freshly boiled and cooled distilled water. Add 0.4 g solid sodium hydroxide and dilute to 1 litre.

8.14 Standard Potassium–iodate Solution (0.025 N)

Dissolve 0.8917 g potassium iodate (KIO_3) in 1 litre distilled water.

9 PROCEDURE

9.1 Cleaning of Equipment

All glass-wares should be thoroughly cleaned. The following procedure is recommended.

9.1.1 Wash with a detergent solution and rinse under tap water followed by distilled water rinsing. Soak in 1 : 1 concentrated nitric acid (*v/v*) for 30 min and then rinse with tap water followed by reagent grade water rinsing.

9.2 Standardization of Reagents

9.2.1 *Standardization of Sodium Thio-sulphate ($Na_2S_2O_3$) Solution (0.025 N)*

Take 25 ml of potassium iodate solution (0.025 N) (**8.15**) in a 250 ml glass-stoppered conical flask. Add 1 g KI to it. Add 5 ml concentrated H_2SO_4 to it. Stopper it and keep in dark for few minutes. Then titrate against standard sodium thio-sulphate solution (**8.14**) taken as titrant using starch indicator. Note the titre value. Determine the strength of $Na_2S_2O_3$ solution from the normality equation.

9.2.2 *Determination of Concentration of Stock Sulphide Solution*

Take 100 ml of distilled water in a 250 ml conical flask and add 20 ml of standard iodine solution (0.025 N) (**8.13**). Add 25 ml of hydrochloric acid (0.1 N) and titrate with the thio-sulphate solution using starch solution as indicator. Note the titrant reading as ‘*A*’.

Take 100 ml of distilled water in another 250 ml conical flask and add 20 ml of standard iodine solution (0.025 N) (**8.13**). Add 25 ml of hydrochloric acid (0.1 N) and 20 ml of the stock sodium sulphide solution

(as prepared in **8.11** above) and repeat the titration with the sodium thio-sulphate solution. Note the titrant reading as '*B*'.

Calculate the concentration of the sodium sulphide solution in terms of H_2S as follows:

Concentration of sulphide solution =

$$\frac{(A-B)\times N\times 17\times 1000}{20}\ \mu g\ H_2S/ml$$

where

N = normality of sodium-thiosulphate solution.

9.3 Sampling

Procedures described are for short-term (30 min or 1 h) and for long-term sampling (8 h or 24 h). One can select different combinations of sampling rate and sampling time to meet special requirements. Sample volumes should be adjusted so that linearity is maintained between transmission and concentration over the dynamic range.

9.3.1 *Short-term Sampling (30 min and 1 h Sampling)*

Place 20 ml of the absorbing solution (**8.10**) in an impinger. Collect the sample at the rate of 1.1/min for 30 min or at 0.5 l/min for 1 h using either the rotameter or a critical orifice device to control the flow. Protect the absorbing reagent from direct sunlight during and after the sampling by covering the impinger with aluminium foil to prevent deterioration. Alternatively, use amber-coloured impingers for sampling. Determine the volume of air sampled by multiplying the flow rate by the time in minutes and record the atmospheric pressure and temperature. After sampling, transfer the sampling solution after shaking to dark-coloured polythene bottles and cap tightly. Transport the bottles to laboratory for analysis. Refrigerate samples if analysis cannot be done within a day.

9.3.2 *Long-term Sampling (8 or 24 h)*

Place 20 ml of the absorbing solution (**8.10**) in an impinger and collect the sample at the rate of 0.2 1/min for 24 h. The applicability of the method to long-term sampling (8 h or 24 h) is subject to calibration at the intended sampling rate and concentration range for the intended period of time. Make sure that no entrainment of solution results within the impinger. During sample collection and storage, protect the sample from direct sunlight. Preferably use amber-coloured impingers for sampling. Determine the total air volume by multiplying the flow rate by the sampling time in minutes. After sampling, transfer the sampling solution after shaking to dark-coloured polythene bottles and cap tightly. Transport the bottles to laboratory for analysis. Refrigerate samples if analysis cannot be done within a day.

9.4 Analysis

Add 1.5 ml of the amine-sulphuric acid standard solution (**8.6**) to the polyethylene bottles and mix. Add 1 drop of ferric chloride solution and mix. Transfer the solution quantitatively to a 25 ml volumetric flask. Discharge the colour due to the ferric ion by adding 1 drop ammonium phosphate solution. If the yellow colour is not destroyed by 1 drop of ammonium phosphate solution, continue drop-wise addition until solution is decolorized.

Make up the volume with distilled water and allow to stand for 30 min. Prepare a reference solution in the same manner using a 10 ml of absorbing solution through which no air has been aspirated and adding the same reagents as added to sample. Zero the spectrophotometer at 670 nm using the reference solution. Measure the absorbance of the sample at 670 nm against the reference solution and note it as '*D*'. After colour development, the methylene blue colour is stable at least for 48 h at room temperature and in the dark. If ethanol is added to inhibit foaming, accelerated decay of colour may occur.

9.5 Preparation of Calibration Graph

Pipet 10 ml of the absorbing solution (**8.10**) in each of a series of 25 ml volumetric flasks and add 0.25, 0.5, 0.75, 1.0, 1.5, 2.0 and 2.5 ml of the diluted sulphide solution (**8.12**) to each of the volumetric flasks. Add 1.5 ml of the amine-sulphuric acid standard solution to each flask and mix. Add 1 drop of ferric chloride solution and mix. Discharge the colour due to the ferric ion by adding 1 drop ammonium phosphate solution. If the yellow colour is not destroyed by 1 drop ammonium phosphate solution, continue drop-wise addition until solution is decolorized. Make up the volume with distilled water and allow to stand for 30 min. Prepare a reagent blank by adding all the reagents as above except the diluted sulphide solution. Zero the spectrophotometer at 670 nm using the reagent blank. Measure the absorbance readings of the sulphide standards against the reagent blank as reference at 670 nm. Plot the absorbance readings against the calculatedconcentrations of the standard solutions to prepare a calibration graph. Determine the slope of the graph.

10 CALCULATION

Hydrogen sulphide concentration = $\frac{D\times k}{V}\ \mu g/m^3$

where

D = absorbance reading of sample against reference solution,

k = 1/slope of calibration curve, and

V = volume of sample, in m^3.

NOTE — 1 $\mu g/m^3$ hydrogen sulphide ~ 0.72 ppm hydrogen sulphide at 25 °C and 760 mm Hg pressure.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Air Quality Sectional Committee, CHD 35

Organization	*Representative(s)*
Bhabha Atomic Research Centre, Mumbai	Dr Gauri Pandit (***Chairman***)
Avantha Centre for Industrial Research and Development, Yamuna Nagar	Dr Nishikant Bhardwaj Dr Sunil Kumar (*Alternate*)
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Indian Oil Corporation Ltd, Faridabad	Dr M. P. Singh
Jharkhand State Pollution Control Board	Shri R. N. Kashyap

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Member Secretary

MS KREETI DAS
SCIENTIST 'B' (CHD), BIS

BIBLIOGRAPHY

a) www.osha.gov/publications/hydrogen_sulfide.html.

This Indian Standard has been developed from Doc No.: CHD 35 (10810).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi